## YARDSTICKS FROM SPACE

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WESTCOTT \*\*\* RULE

## Microscopic plastic beads have become the newest standard in measurement

By JANET RALOFF

he maiden voyage of the space shuttle Challenger marked the resumption of spacewalking by U.S. astronauts after a nine-year hiatus and the deployment of the first U.S. tracking and data-relay satellite (SN: 4/16/83, p. 244). What is less well known is that the April 1983 flight also produced the first commercial product "made in space." Last month, billions of the tiny plastic spheres manufactured in the shuttle's cargo bay during that flight went on sale as the National Bureau of Standards' (NBS') newest measurement standard.

These little beads, each a near-perfect 10 microns in diameter (9.89 microns, actually, give or take 0.04), can be thought of as microscopic "yardsticks." To a carpet installer, a yardstick or tape measure helps avoid waste in cutting expensive carpeting and provides for its snug, wall-to-wall installation. "The need for a tape measure in the microscopic world is just as real, only it's a little harder to visualize," says Stanley Rasberry, chief of NBS' Office of Standard Reference Materials in Gaithersburg, Md.

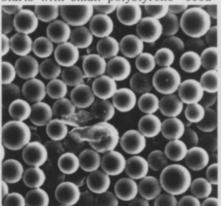
Researchers working on the project anticipate that these tiny polystyrene beads will be used in the calibration of tools that measure microscopic particles in ground powders — be they grains of cake flour, face powder, paint pigment or the silver used in photographic emulsions. Alternatively, they can be used to gauge the size of human blood cells or of pollutant particles filtered from air.

While the beads are unquestionably small — 18,000 of them would fit on the head of a standard dressmaker's pin —

they are 30 times larger than the smallest polystyrene spheres that NBS now offers as commercial reference standards. In fact, it's not how small but instead how relatively *large* they are that has made reproducible reference standards in the 10-, 30-, 60- and 100-micron range impossible to manufacture. Physics works against scaling to these larger sizes — on earth, that is.

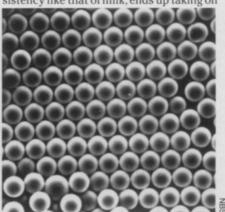
Since it's gravity that tends to gum things up, chemists at Lehigh University in Bethlehem, Pa., decided to try manufacturing the beads out of this world. And NASA was only too happy to oblige, offering them free space aboard each of five different shuttle flights in the hope of building up a commercial market for its launch services.

The new reference spheres were made in a process somewhat analogous to swelling microscopic sponges with water. It starts with small polystyrene "seed"



spheres suspended in water. A measured quantity of liquid styrene monomer — individual styrene molecules—is mixed into this stable suspension of small hydrocarbon particles in water, a latex. As the monomer penetrates the seeds, they swell. To retain their inflated size, the spheres are than "baked" in a reactor. Chemicals in the reactor link the spheres' monomer molecules into chains (polymers) of styrene, changing the liquid spheres into solid beads. Through a series of swelling/baking phases, the Lehigh team grew 10-micron spheres from what were initially 0.24-micron seeds.

Because the monomer is lighter than water, when the swelling phase is conducted on earth the inflating spheres become increasingly buoyant. Rising to the top of the latex, they quickly agglomerate into clumps that distort each sphere's shape. The latex, which began with a consistency like that of milk, ends up taking on



Tiny space spheres (right) are rounder and more uniform than earthly counterparts (left).

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the consistency of something closer to cottage cheese. Baking poses a similar distortion risk, because as the spheres solidify, they become denser. Sinking to the bottom of the reactor, they again bump together, sticking into clumps. And even those that survive the clumping tend to take more of an egg shape than a ball shape.

Though stirring the latex can sometimes alleviate clumping, "these particles, once swollen with styrene monomer, are soft and sticky," explains John Vanderhoff, co-director of Lehigh's Emulsion Polymers Institute and a project leader for the space manufacturing project. "So if you stir the latex too hard," he says, "the soft, sticky particles will come together, clot and form cottage cheese." In a high-gravity environment, the larger the particles, the greater the need for stirring. "For a 100cubic-centimeter reactor on earth we would use 100 to 400 revolutions per minute," he says. "In some of our space experiments we've used only 3 rpm." The slower stirring necessary in space means that relatively large particles don't coagulate

The result is that the beads produced in space are not only more spherical than those manufactured on earth but also more uniform in size. Uniformity is particularly important in manufacturing standard reference materials. Since it's impossible to measure each of the billions of beads produced in any batch, all must be nearly the same size so that measurements of a few will prove characteristic of them all.

As keeper and purveyor of the nation's most precise measurement standards, NBS was called in to certify both the size and the uniformity of the new space spheres. The task required development of a new imaging technique having a resolution approaching that of electron microscopy. Called center distance finding, it uses an optical microscope to view the transparent beads, illuminated with collimated (parallel) light.

Each microsphere to be measured must touch another, explains the technique's developer, Arie W. Hartman of NBS. To achieve that, a suspension of spheres in water is smeared onto a microscope slide and allowed to dry. Surface-tension forces of the shrinking liquid pull the spheres into compact groups. Because each sphere is transparent and acts as a refracting lens, he says, when illuminated with collimated incandescent light the spheres focus that light into points. On a photograph, each point marks the center of a sphere. The distance between any two that touch represents the sum of their radii or the diameter of each, if the spheres are

NBS is charging \$384 for each 25-gram vial containing about 30 million of the 10-micron spheres. The quarter-million dollars it expects to recover when all are sold "is clearly not enough to completely

Microscope objective

Microscope slide

Microscope slide

Focal spots

Before viewing, 10-micron beads are pulled into touching clumps (lower left). Under illumination with parallel light, each bead focuses light to a point (lower right). For

uniform spheres, the distance between two that touch equals a bead's diameter.

remunerate NASA for [their] production," Rasberry says. It is, however, roughly twice what NBS spent to certify their size, so NBS will turn over half its proceeds to NASA as partial compensation for the space agency's costs.

Thirty-micron beads made aboard the most recent sphere-manufacturing shuttle flight, in February 1984, were turned over to NBS last June for size certification. NASA's Marshall Space Flight Center in Huntsville, Ala., directed the manufacturing project. These 30-micron beads, says Jack Lee, deputy director of the Marshall Center, "could very well become the second space product to be sold commercially." On three future, but as yet unscheduled, missions even larger beads will be developed. "By the end of the project we hope to produce 100-micron particles to go along with the 10- and 30-micron beads we've already produced," Lee says.

Vanderhoff has already formed a company, Particle Technology, Inc., to commercialize the manufacturing process. And he sees a valuable market developing for his microscopic products. The reference spheres' value, he notes, lies not in what they're made from — a synthetic resin used in everything from Styrofoam and clear plastic drinking cups to molded household goods and air conditioner cases — but in their size. "Polystrene," explains Vanderhoff, "currently sells for

around 50¢ a pound, but calibration particles, very narrow in size distribution and carefully measured, will bring between \$2 million and \$7 million per pound."

His goal is to increase production so that he retrieves about 1.3 pounds of polymer per flight. Once the commercial value of the manufacturing process is established, NASA plans to begin charging Vanderhoff for his use of space and launch services — much in the way it charges commercial satellite makers to launch today.

The microspheres are "the first of what we expect will be a long line of products to carry a made-in-space label," NASA Administrator James M. Beggs said in announcing their initial sale. Referring to NASA's plan for an orbiting space station, Beggs said that "by the early to mid-1990s we will have a permanent presence in space," opening "boundless opportunities, not only for commerce and industry but also for other advances in science and technology. Businesses large and small are beginning to see the potential for the station as an industrial research laboratory and space factory."

Among the manufacturing areas he foresees being exploited in space are production of pure pharmaceuticals, perfect crystals larger than any made on earth, thin films for electronics applications and new alloys for industry.

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